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Surface preparation processing for superconducting cavities*

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Surface preparation is an important processing in production procedures of superconducting niobium cavities, deciding whether the performance of the niobium cavities can meet the specifications. A series of surface preparation methods and relevant apparatuses have been constructed at Shanghai Institute of Applied Physics (SINAP) and the standard procedures of cavity processing were established and successfully applied to different types of cavities. With standard surface preparation procedures on the 500 MHz 5-cell niobium cavity, the cavity accelerating voltage at $T = 4.2\,\mathrm{K}$ reached 7.5 MV while its quality factor was still higher than 1×10^9. The accelerating gradient of the IMP-HWR010 cavity reached 4.9 MV/m with the quality factor of better than 3×10^8 at 4.2 K.

Keywords: Surface preparation, Barrel polishing, Buffered chemical polishing, High temperature baking, High pressure rinsing

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I. INTRODUCTION

Superconducting (SC) niobium cavity is a key component for accelerating charged particles to compensate for beam loss or to hit the target material. Its accelerating field gradient can reach 50 MV/m with a little power dissipation [1]. With a high Q-value, the high gradient performance of an SC niobium cavity is sensitive to surface imperfections, such as damaged layer, scratches, inclusions, electron beam welding defects, and contamination. Each may cause cavity loss mechanisms such as field emission or thermal breakdown [2]. Cleanness of the inner surface of SC cavity is an indispensable condition for successful performance of the accelerating structures, hence the necessity of surface preparation for an SC cavity to eliminate such problems. The methods for surface preparation of niobium cavities include barrel polishing [3], buffered chemical polishing (BCP) [4, 5], electropolishing (EP) [6], high pressure rinsing (HPR) [7, 8], low or high temperature baking [9] etc.

A 500 MHz 5-cell niobium cavity was fabricated at the Shanghai Key Lab of Cryogenics & Superconducting RF Technology of Shanghai Institute of Applied Physics (SINAP) [10, 11]. Barrel polishing treatment was used as a primary procedure mainly due to its effective process technique and lower cost. Bulk chemical polishing, high temperature baking and slight chemical polishing treatments were then implemented in sequence. HPR was used as the final step of cavity preparation to reduce field emission [7]. Also, surface preparation for the cavity of a 162.5 MHz $\beta = 0.10$ niobium half-wave resonator at the Institute of Modern Physics, Chinese Academy of Sciences (IMP-HWR010), was undertaken at SINAP. The cavity went through a series of surface treatments, such as degreasing, bulk chemical polishing, high temperature baking, slight chemical polishing and ultrasonic rinsing with ultra-pure water. The enormous internal cubage and surface area of the 500 MHz 5-cell niobium cavity, and the complicated structure of HWR cavity, were the most important considerations for surface preparation processing. In this paper, standard procedures of surface preparation processing and results of the radio frequency (RF) vertical test for the two types of niobium cavities are reported.

II. CAVITY SURFACE PREPARATION SEQUENCE

A. Degreasing and barrel polishing

It is well known that a rather thick niobium layer of 150 μm (usually so called damaged layer) should be removed from the inner surface during cavity preparation, so as to achieve a high accelerating gradient. Barrel polishing, which is often called tumbling for cavities, is a kind of mechanical grinding, rotating the cavity with chips, water and compounds. Barrel polishing can partly replace chemical or electrochemical polishing, which is costly and environmentally critical. In addition, a fairly smooth surface after barrel polishing is a good condition to start chemical polishing. Buffing was used to each half cell of 500 MHz 5-cell cavity before electron beam welding, and barrel polishing was adopted for the completed structure. This method is quite convenient for multi-cell structures, being capable of grinding the whole inner surfaces including the welding seams.

The barrel polishing apparatus was installed at SINAP

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Fig. 1. (Color online) Barrel polishing apparatus for the 500 MHz 5-cell niobium cavity.

for the 500 MHz 5-cell prototype cavity (Fig. 1). It has two pedestals connected by four stands. The frame is rotated by the main motor under controllable speed between 0–200 rot/min. The 5-cell cavity was filled with 200 kg of grinding chips with plastic binding added with 150 kg water and 1.5 kg compound. The cavity, covered tightly and fixed in the lodgment, was rotated around its axis in speed of 40–50 rot/min. After 163 h of barrel polishing, a layer of 90–100 μm was removed, which is sufficient for complete removal of all protrusions, scratches and other surface defects. Also, the barrel polishing rendered a preparatory smoothing of the welding areas. Barrel polishing was not done with the IMP-HWR010 cavity. It was just degreased and cleaned using acetone and alcohol separately.

B. Bulk chemical polishing and rinsing

As mechanical grinding itself resulted in a damaged layer or contaminated surface by the abrasives, the surface should be refinished with stress-free and contamination-free preparation techniques like chemical polishing, high pressure rinsing etc. Buffered chemical polishing, a convenient surface preparation method, has been used so far as a main preparation technique for SC niobium cavities at SINAP. For the 500 MHz 5-cell niobium cavity, dunk chemistry was awkward because of the large volume of acid required. In this case, a cavity surface closed-loop chemical preparation system was developed. The BCP system (Fig. 2) mainly consists of acid circulating loop, cooling-water circulating loop, low pressure water rinsing pipeline and recovery pipeline of waste acid. The operators are carefully secured against exposure to acids and noxious fumes.

The two cavities were assembled into the closed-loop BCP apparatus. A total of 400 L acid solution containing 1 part HF (40%), 1 part HNO_3 (65%) and 2 parts H_3PO_4 (85%) by volume was pre-mixed in the acid tank and cooled by iced flowing water pipe inside the acid tank to control the acid temperature at 10–15 °C. Next, the acid solution was pumped

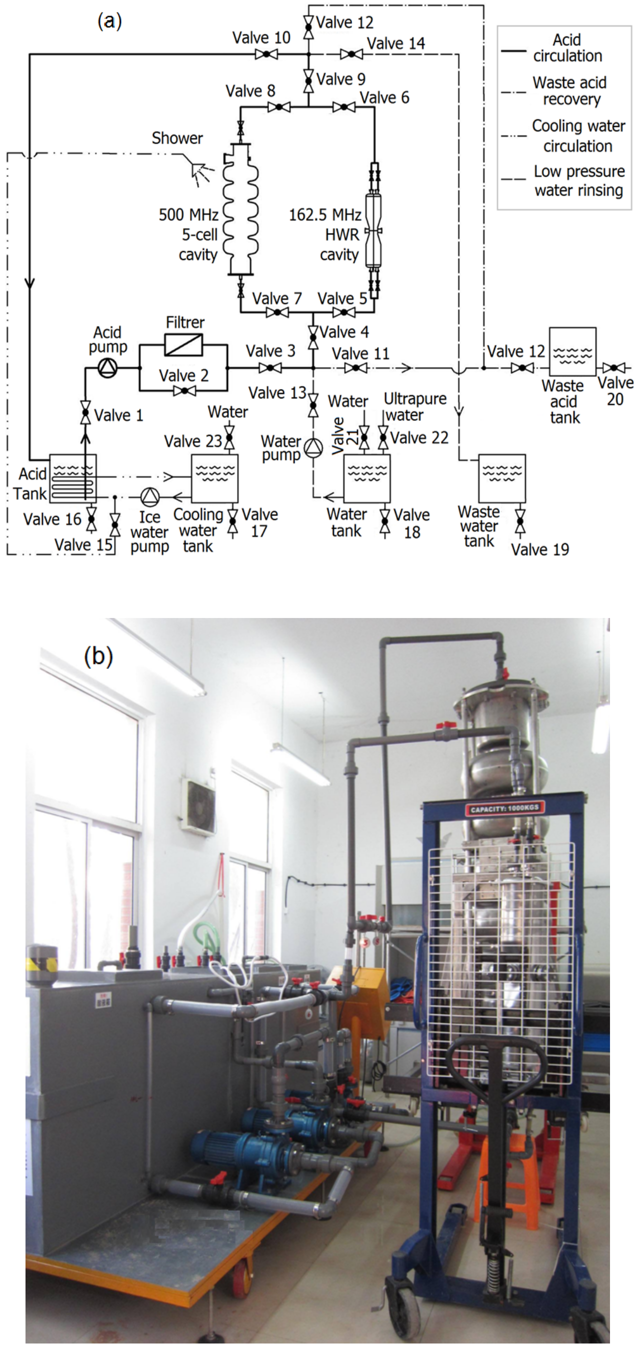

Fig. 2. (Color online) Layout of chemical etching system (a) and apparatus (b) for the two cavities.

through a filter, sent to the IMP-HWR010 cavity, and flowed back to the acid tank; while the valve before the 5-cell niobium cavity was closed. The acid filling time to the cavity corresponding to acid pumping speed was optimized to avoid a noticeable shape distortion of lower part of the cavity by excessive exposure to acid. Iced water showers to the outer of cavity were used for cooling the cavity. Temperatures of the acid solution and cavity itself were well controlled to achieve an ideal and stable etching speed. Then, the acid pump was

switched off, and the acid solution was dumped from the cavity when the damaged surface layer (deep enough) was removed. Finally, low pressure water rinsing was immediately carried out to take out the residual acid on inner surface, lasted for 30 min. A 100–110 μm layer of niobium was removed from inner surface of the IMP-HWR010 cavity in 80-min BCP treatment. The cavity was dismounted and placed into a moveable class-100 clean-room for 2 h ultrasonic rinsing using ultra-pure water.

The 5-cell cavity was treated with a similar chemical polishing process: 80-min BCP treatment and 60-min low-pressure water rinsing. A 90–100 μm layer of niobium was etched from the inner surface. The difference of etched thickness between the two cavities was due to different diameter of the pipes connected with them, leading the different velocity of acid flow in the cavities.

To remove thoroughly chemical residues or particles against field emission, the automated HPR system, consisting of a high-pressure pump, a spray wand, and custom spray nozzles, was applied as the immediate step in the surface preparation of the 5-cell niobium cavity. The ultra-pure water in resistivity of 18 MΩ cm and pressure of 80 kg/cm^2 was sprayed from 6 nozzles on top of the feeding cane, which moved up and down inside the rotating cavity. Finally, the cavity was dismounted and moved into a class-100 clean-room for 2-h HPR, followed by drying in a class-10 clean room for 24 h.

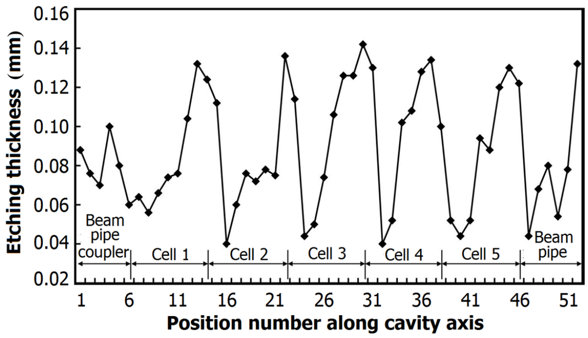

Fig. 3. Material removal distribution along the cavity axis of the 5-cell cavity.

The distribution of removed thickness from the 500 MHz 5-cell cavity, as shown in Fig. 3, was obtained with an ultrasonic thickness meter measuring the cavity wall thickness along the axis. It can be seen that the etching thickness was obviously larger at cavity iris sections. The etching speed was lower at the cavity equator sections in vertical BCP came. A barrier of etched niobium and bubble could buffer further reaction of the acid with the niobium surface. How to acquire a uniform etched thickness in multi-cell cavity will be an issue for further studies.

C. High temperature baking

It is well known that cavities of high purity niobium pick up hydrogen during bulk chemical polishing or electric polishing, and result in the hydrogen Q-disease. The hydrogen precipitates to niobium hydro-ride during slow cooling down between 150 K and 90 K [9] in cold test. It is a weak superconductor, with remarkably increased surface residual resistance of the cavities. Since fast cool-down is difficult in accelerator complex, one has to eliminate the problem before the final cavity assembly. Hydrogen in niobium bulk is easily degassed at above 600 °C. The two cavities were put into a titanium box and heated to $\sim$ 680 °C in a vacuum furnace of better than 1×10^{-3} Pa. The degassing lasted for three hours. Fig. 4 shows the baking temperature and vacuum pressure. Although the baking temperature was too low to have titanium solid getter [12], it is safer to use titanium box with niobium cavities to prevent material degradation from residual gas in the furnace.

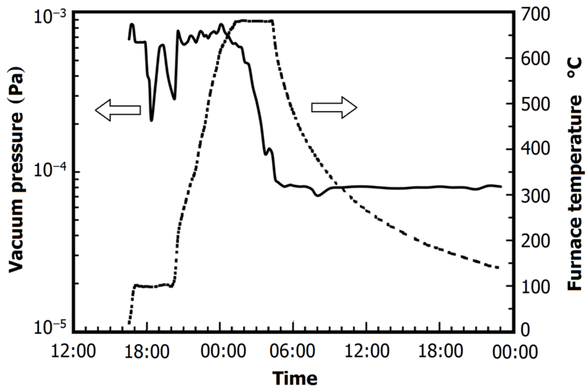

Fig. 4. Baking process for the two cavities.

D. Slight chemical polishing and rinsing

To eliminate titanium or other contamination caused by high temperature baking, a slight chemical polishing was necessitated. Another total of 400 L fresh acid solution was remixed. The IMP-HWR010 cavity was treated with 20-min BCP and 50-min low-pressure water rinsing, while the 5-cell cavity was with 40-min BCP and 90-min low-pressure water rinsing. The cavities were moved into a class-100 clean-room for 2 h ultrasonic and HPR rinsing, respectively, followed by drying in a class-10 clean room for 24 h. It should be emphasized here that vertical positions of the two cavities in BCP apparatus were reversed this time, so as to minimize the inequality of the etching thickness caused by the cavity geometry as far as possible.

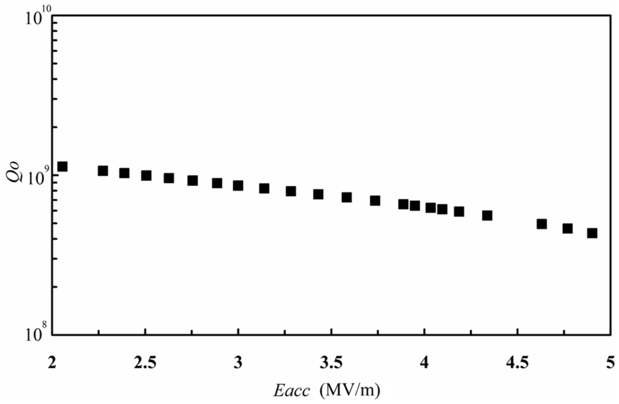

Fig. 5. Q_0 versus accelerating field curve at 4.2 K for the IMP-HWR010 niobium cavity.

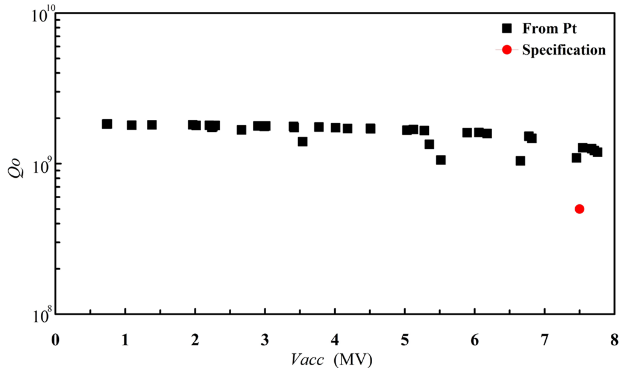

Fig. 6. (Color online) Q_0 versus accelerating voltage curve at 4.2 K for the 500 MHz 5-cell niobium cavity.

E. Assembling and "in-situ" baking

With proper chemical etching and high pressure rinsing, a proper assembling in the clean room was necessary to avoid defects and field emission sites in the cavity. Sealing flanges, low-power input couplers and pickup antennas were mounted on the 5-cell and IMP-HWR010 cavities. The assembled cavities were evacuated gradually with a dedicated pumping system, and leak checked outside the clean room immediately to confirm leak tighten at room temperature. After evacuating and successful leak check at room temperature, the niobium cavities were mounted onto the vertical test stand and followed by "in-situ" baking process under ultrahigh vacuum at 120 °C for 48 h, as the final preparation of niobium cavities. The vacuum of cavities was kept better than 1×10^{-6} Pa.

III. RESULTS OF RF VERTICAL TESTS

The main purpose of RF vertical test [13, 14] was to obtain the cavity accelerating performance and verify whether the procedures and treatments adopted during the fabrication, electron beam welding and surface preparation can meet the requirements or not. The vertical tests were carried out in a vertical cryostat filled with liquid helium at 4.2 K. The measured quality factor Q_0 (unloaded Q value) of the IMP-HWR010 cavity as a function of accelerating field gradient ($E_{\rm acc}$) was shown in Fig. 5. The IMP-HWR010 cavity achieved accelerating field gradients $E_{\rm acc}$ of $\sim$ 4.9 MV/m without quench and quality factor Q_0 above 3×10^8

Figure 6 showed the vertical test result of 500 MHz 5-cell cavity, in which the cavity accelerating voltage reached as high as 8 MV while the quality factor Q_0 was still better than 1×10^9.

IV. CONCLUSION

The surface preparation is a critical step of SC niobium cavities fabrication. Barrel polishing machine, closed-loop BCP apparatus system, high temperature vacuum furnace and ultra-pure water HPR system were constructed at SINAP, and applied successfully to the 500 MHz 5-cell SC cavity and IMP-HWR010 cavity. The standard procedures of cavities processing and handling can be summarized as follows:

- Degreasing and cleaning using acetone and alcohol separately;
- Barrel polishing around 100 µm;
- Bulk chemical polishing around 90–110 µm in 80 min, and 1 h low-pressure water rinsing;
- Rinsing with ultra-pure water (18 MΩ-cm and 8 MPa) for 2 h or ultrasonic rinsing using ultra-pure water;
- 680 °C annealing for 3 h to remove hydrogen during bulk chemical polishing;
- Slight chemical polishing around 15–30 µm in 20 $\sim$ 40 min, and 1.5 h low-pressure water rinsing;
- HPR for 2 h or ultrasonic rinsing using ultra-pure water;
- Drying in a class-10 clean room for 24 h;
- Assembling in the class-100 clean room and leak checked;
- "*In-situ*" baking process under ultrahigh vacuum at 120 °C for 48 h.

The two types of niobium cavities went through the sequence of standard surface preparation processing, the IMP-HWR010 cavity had exceeded an accelerating gradient of 4.9 MV/m with quality factor $Q_0 \geq 3 \times 10^8$ at 4.2 K, and the 500 MHz 5-cell cavity had exceeded accelerating voltage of 7.5 MV with a quality factor $Q_0 \geq 1 \times 10^9$.

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